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Dpermeation by zeolite NaA membranes

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Zeolite NaA membranes have been industrialized for pervaporative dehydration over ethanol and isopropyl alcohol because of the high hydrophilicity and molecular sieve of zeolite layers. Zeolite NaA membranes were normally prepared on porous ceramic (alumina and mullite) supports, especially for the reports of the scale-up synthesis. In this study, we reported the scale-up preparation of high-flux NaA membranes by a unique synthesis device on porous stainless steel tubular supports and the pilot dehydration of ethanol via vapor permeation process.

Zeolite NaA membranes were scaled up using a large autoclave that can contain 50 pieces of 100-cm-long tubes. The large autoclave, which has a volume of 50 liters and a diameter of 20 cm, were heated by resistant heating rods in the centre and heating jacket with following hot oil. Fifties membranes were distributed on two circles with the diameters of approximately 12 cm (20 tubes) and 16 cm (30 tubes), respectively. The temperature drop could be controlled within 5 ˚C at synthesis temperature of 120 ˚C by adjusting the two heating ways. Compared with our small autoclave (4.5 cm ID), the synthesis in the large autoclave became more effective, energy-saving and 25 % gel-saving. One hundred and sixty membranes were prepared using the large autoclave for 7 batches (100 membranes for 2 batches and 60 ones for 5 batches). As a result, One hundred and fifty-two membranes were successful. Those membranes showed an average flux of 6.0 kg/m^2.h and an average separation factor of 9000 for a feed of 10 wt% water/ethanol mixture at 75 ˚C. Analysis of the membranes by SEM indicate that the thickness and crystal morphologies of zeolite NaA layers are uniform and independent of the location in the autoclave. The typical membrane surface SEM image was showed in Fig.1. The stainless steel support used for membrane synthesis could be recovered by washing in high NaOH base solutions at round 130 ˚C. The membranes using the 4-times-refreshed supports still showed high pervaporation performance, which showed the stainless steel showed potential application for recovery and reuse to save the cost of membrane preparation.

A 500 ton/year pilot vapor permeation set-up was built by Misui Engineering & shipbuilding Co. Ltd. (MES) installed our membranes for ethanol dehydration from 93 wt.% to 99.8 wt.% (shown in Fig. 2). Three membrane modules were connected to keep the dehydration effective. The design of membrane modules were described in the reference [1]. Each module contained 31 1-meter-long zeolite NaA membranes and had a total membrane area of 1.1 square meters. The water/ethanol feed with flow rate of 69.4 kg/h (equal to 500 ton/year) was through a heater to increase its temperature from room temperature to 120 ˚C. The vapor continuously went through the three membrane modules to form fuel ethanol. The average flux for the total membrane in three modules was up to 1.37 kg/m^2.h for the vapor permeation process (ethanol from 93 to 99.8 wt.%) at 120 ˚C. The set-up has been running for over 3 months with good stability. The energy consumption and operation cost for the set-up for the dehydration of ethanol were estimated. The average energy consumption of every ton fuel ethanol was calculated as 380 kwh based on the 3 months' test for the set-up. The cost of membrane change was estimated to be around 10 £/ton ethanol based on the assumption of 2.5 years' lifetime similar to the normal industrialized zeolite NaA membranes. High flux of the membrane deceased both the investment and operation cost.

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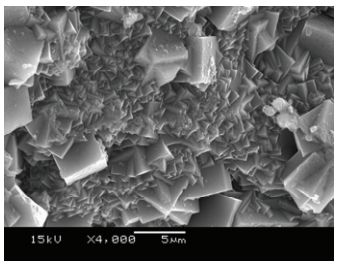

Fig.1 surface SEM image of zeolite NaA membranes

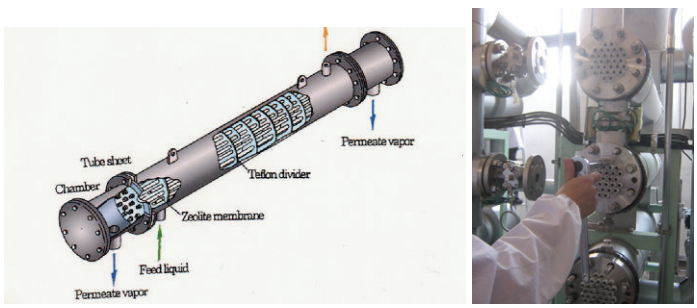

Fig.2 smembrane modules (left) and the dehydration setup (right)

Reference

1. Morigami Y, Kondo M, Abe J, et al, The first large-scale pervaporation plant using tubular-type module with zeolite NaA membrane, Sep. Purif. Technol., 2001, 25 (1-3), 251-260.

Keywords: Zeolite Membrane, Scale up, Dehydration, Vapor Permeation